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The spring constant calibration of the piezoresistive cantilever based biosensor

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Abstract

Piezoresistive microcantilevers are widely applied to measurements of low forces, masses and viscosity [1]. After surface functionalization they might be used as a biochemical sensors being capable of the intermolecular force investigation. The problem is that such sensors change its mechanical properties in the environment they operate. Therefore there is a need for a high accuracy technique being capable of measuring of mechanical properties of functionalized cantilevers operating in the target environment. We suppose that such conditions meet the analysis of thermomechanical oscillation noise.
In this paper the analysis of two types of cantilevers, that might be used in bioelectrochemical measurements, was performed. We determined the cantilever deflection and force sensitivity. The spring constant was measured by three different methods: the Cleveland methods [2] and the thermomechanical noise analysis. The obtained results indicate that analysis of thermomechanical excitation noise is the simplest and the most reliable method for spring constant calibration of piezoresistive cantilever based sensors.

Keywords: biosensor, piezoresistive microcantilever, calibration

1. Introduction

Piezoresistive microcantilevers are attractive devices for bioelectrochemical sensing due to easy to handle electric detection of the cantilever deflection [1]. Moreover, an electrical readout enables measurements in nontransparent environments (e.g. blood) and creates excellent opportunity for miniaturization [2]. Additionally piezoresistive microcantilevers might be combined into an array and function as an electronic nose [3]. The drawback of this solution is that piezoresisitve cantilever based bioelectrochemical sensors are usually complex devices for which it is difficult to determine its mechanical properties. Moreover as biosensors they operate in liquids which drastically changes their mechanical properties. From that reason piezoresistive cantilevers are used for detection rather then measurements. These circumstances would change if the proper calibration procedure were found. Such procedure

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should be able to determine the cantilever spring constant without any assumption about the cantilever structure and the target environment. From among calibration procedures developed up to know only methods based on loading by known force [4] and on thermal noise analysis [5] meet conditions presented above. The thermo-mechanical noise might be registered in any environment and it is valuable source of information about cantilever mechanical properties. This information might be exploited by careful analysis.

For cantilever with optical detection Pierzer and Hugel [6] show that proper analysis of the thermal oscillation noise in viscous liquids enables determination of spring constant with uncertainty about 10%. Up to know the thermal noise analysis was applied to cantilevers with optical detection, in case of which registration of thermal noise is much easier, due to the greater deflection sensitivity of optical setup.

In this paper we utilize thermal method for the spring constant calibration of piezoresistive microcantilevers. Next we compare the results with methods based on micromass loading and geometrical dimensions. The novelty of our research relies on the fact, that such comparison of spring constant calibration methods for piezoresistive cantilevers was not up to know presented.

2. Methodology

In order to determine the sensitivity of the piezoresistive detector the microcantilever is bended by precisely measured distance (Fig. 1). Simultaneously the signal from piezoresistive readout is registered. The sensitivity is computed as a voltage to deflection ratio. The deflection is induced by the tungsten tip excited into the vibration by means of the piezoelement mounted to the second end of the tungsten wire. The microcantilever deflection is measured very precisely by means of a Fabry-Perot interferometer. Simultaneous measuring of the voltage signal on the output of the piezoresistive deflection detector of the microcantilever enables computation of the microcantilever sensitivity.

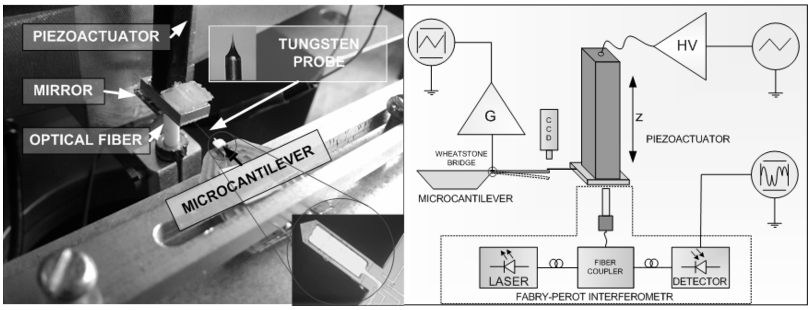

Fig. 1. The measurement system for deflection sensitivity calibration

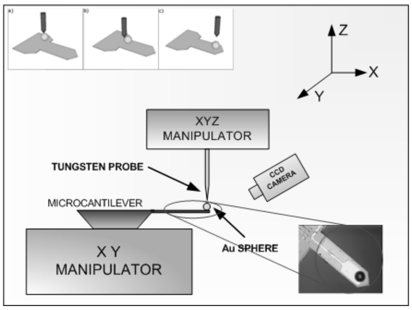

Fig. 2. Micromanipulator system for micromass mounting

In order to register thermomechanical noise of the piezoresistive microcantilver the high gain amplifier is necessary. We constructed such preamplifier and performed necessary measurements of its transmittance to determine its noise properties. We used high frequency, low noise components to construct the measurement electronics.

The preamplifier output is connected to the input of PCI 6251 National Instruments data acquisition card and the signal is analyzed by means of the software designed in the LabView programming environment. The software enables power spectrum estimation and fitting by means of the simple harmonic oscillator (SHO) model given by equation

$$S_T(f) = A/[(f_R^2 - f^2)^2 + (f_R f/Q)^2] \tag{1}$$

The estimated parameters (A, f_R – resonance frequency , Q – quality factor) enable calculation spring constant basing on the formula.

$$k_T = 2k_B T f_R / [\pi A Q\ ] \tag{2}$$

Another calibration method is based on micromass loading. It is capable of determining of a spring constant without any assumptions on a cantilever structure but it can not be utilized in the biosensor target environment. We apply this method to verify results obtained by means of the thermal method. Mass loading method relies on the fact that the reference micromass mounted at the end of the investigated microcantilever leads to resonance frequency shift. The shift might be utilized for the determination of the spring constant on basis of equation

$$k_M = (2\pi)^2 m / (f_{R2}^{-2} - f_{R1}^{-2}) \tag{3}$$

where m- is a micromass placed at the cantilever end, f_{R1}, f_{R2}- resonance frequencies of the unloaded and loaded cantilevers respectively. The most difficult part of this method is the process of mass mounting. Microcantilevers are very delicate structures that might be easy destroyed during this procedure. We use golden spheres as a reference masses. Their radiuses are measured by means of the optical microscope. On the basis of gold density and sphere volume the mass might be determined. The micromass is lifted up from the glass surface with the help of adhesion forces. If the tungsten tip touches the golden sphere the gold-tungsten adhesion forces attaches it to the tip. Next the sphere is put on the microcantilever base and after that slowly rolled at the cantilever end.

If the cantilever has simple uniform structure, it is also possible to compute spring constant on the basis of the resonance frequency and geometrical dimensions of an unloaded microcantilever. This method utilizes following formula

$$k_F = 2(\pi l f_R)^3 w \rho^{3/2} E^{-1/2} \tag{4}$$

where l – is cantlever length, w – width, ρ – mass density and E – Young modulus.

3. Results

The measurements were made on 600 μm long and 157 μm wide piezoresisitve cantilevers designated for bioelectrochemical sensing. The investigated microcantilvers are coated by golden layers of different areas (Fig. 2). The obtained results are collected in table 1.

Table 1. The measured parameters of the investigated cantilevers

Parameters	Spring constant k_T (thermal noise)	Spring constant k_M (mass loading)	Spring constant k_F (dimensions and resonance frequencncy)	Deflection sensitivity	Force sensitivity	Thermal fluctuation force in 1 Hz bandwidth
Units		[N/m]		[μV/nm]	[μV/nN]	[fN]
Cantilever A	0.65±0.05	0.7±0.2	1.4±0.3	1.04±0.01	1.6±0.1	82
Cantilever B	5.7±0.2	6.0±0.9	9.4±0.3	4.52±0.01	0.79±0.02	35

The deflection sensitivities were measured while the Wheatstone bridge bias was 5 V.

Next we registered thermomechanical oscillation noise. Fitting SHO model to the power spectra (Fig. 3) enables determination of spring constant value k_T with relative error equal to 8%. We measured the resonance frequency of unloaded cantilevers and by means of the micromanipulator system we mounted the microspheres on the cantilevers ends.

After cantilever loading spectral analysis revealed resonance frequency shifts (Fig. 3be). On the basis of these shifts the spring constant values k_M, were calculated. Additionally, we use the cantilevers geometry measured by means of an optical microscope and the value of resonance frequency of the unloaded microcantilever to evaluate the spring constant values k_F. In order to evaluate this value we assume that cantilever is made off silicon (ρ=2.329 g/cm^{-3}, E=185 GPa).

We use k_T values to compute the force sensitivity and the thermal fluctuation force. The fluctuation force might be interpreted as the minimum detectable force in the microcantilever resonance operation mode .

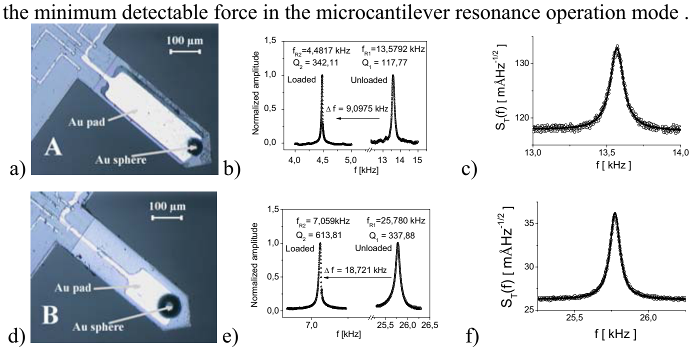

Fig. 3. Investigated biosensors: a,b) optical images of loaded piezoresistive microcantilevers; b,e) corresponding frequency shifts; c,f) noise power spectra of unloaded microcantilevers and simple harmonic oscillator model fits

4. Concluding remarks

In this article, for the first time for piezoresistive microcantilevers, three different spring constant calibration methods (thermal noise method, added mass resonance method, unloaded frequency method) have been compared. The obtained results show that the analysis of thermomechanical oscillations provides the best estimates of spring constant value (the least relative error). The k_M confidence intervals contain k_T confidence intervals. The results show that k_F value is strongly biased although its confidence intervals are quite narrow. We supposed that this bias is caused by oversimplified assumption about the cantilevers structures. The interesting fact is that cantilever A with almost 5 times worse deflection sensitivity has 2 times better force sensitivity. It shows how extremely important, for force measurements, the calibration of a microcantilever spring constant is.

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